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Thermoluminescence, Optical, and IR Spectroscopic Properties of the Li2O–CaO–MgO–B2O3 Glass System for Gamma-Ray Dosimetry

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ABSTRACT: In this work, borate glass samples with the composition xMgO–10Li2O–10CaO–(80–x)B2O3, (x= 5–25 mol% MgO) were fabricated and evaluated for their suitability for radiation dosimetry. The glass samples were synthesized using the melt-quenching technique. The starting components were melted at 1200 °C for 1 h, followed by annealing at 350 °C for 2 h to relieve internal stresses and prevent cracking. The samples were prepared as discs with a diameter of 3 mm and a thickness of 1 mm for thermoluminescence measurements and were subsequently irradiated using gamma rays from a Co-60 source. The effect of MgO content on the physical and optical properties was investigated using X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, and UV–visible absorption spectroscopy. XRD analysis confirmed the amorphous nature of the fabricated glasses, while FTIR spectra revealed the presence of both BO3 and BO4 structural units. UV–visible spectra showed a decrease in the indirect optical band gap with increasing MgO content. The prepared samples were irradiated with different gamma doses, among which the sample containing 5 mol% MgO exhibited the highest integrated TL intensity. Dose–response and fading studies were also carried out, demonstrating improved linearity and low fading. These results suggest that the lithium borate–calcium glass system modified with 5 mol% MgO may be suitable for radiation dose measurement.

1. INTRODUCTION

Thermoluminescent dosimeter (TLD) are widely used for monitoring personal radiation exposure in industrial, medical, and research settings.[1] TLDs absorb and store energy from ionizing radiation, then release it as light when heated. Their key advantages include sensitivity, reusability, and tissue-equivalence.[2,3]

Common TLD materials include CaF_2, LiF, and $CaSO_4$. LiF is widely used due to its density being close to that of human tissue.[4] The search for alternative glass-based TLD focuses on materials with tunable sensitivity, improved tissue equivalence, and stable thermoluminescent properties.

Borate glasses are widely used because of their promising properties, including low melting points, high optical transparency, excellent thermal stability, and the ability to dissolve rare earth ions.[5,6] Glasses containing boron, such as lithium borate glasses, exhibit unique electrical properties. Depending on the type of transition metal oxide (TMO) dopants incorporated into the glass

matrix, they can function as either dielectrics or insulators.[7] Furthermore, lithium borate glasses doped with TMO showed promise in a variety of applications. Due to their semiconducting characteristics, they can also be used as cathode materials in lithium-ion batteries.

Doping the Li_2O-B_2O_3 glass system with silver atoms (105 ppm) enhanced the glow peaks at approximately at 220°C and 320°C and resulted in a stronger thermoluminescence (TL) response compared with the undoped glass. The dosimeters also exhibited a linear gamma-rays dose response over the range of 1-70 Gy.[8]

Lithium works as a network modifier by establishing ionic interactions with oxygen, resulting in non-bridging oxygen (NBO) sites. This minimizes the hygroscopicity of borate glass while increasing its structural solidarity. These modifications can also lead to the formation of color centers.[9] Furthermore, due to its high ionic conductivity, lithium borate is suitable for infrared and laser-related applications. Its effective atomic number (Z_{eff}) is similar to that of human tissue, making it a promising material for medical radiation dosimetry.[10]

Fluorite (CaF_2) can be added to B_2O_3-based glasses, yielding several beneficial effects. One of the main advantages is the reduction of the melting point, which enhances the glass-forming process and allows for more efficient synthesis.[11] Additionally, CaF_2 improves optical clarity, making the glass more transparent and suitable for applications requiring high optical performance. This structural modification can also enhance physical properties, such as thermal stability and mechanical strength, depending on the composition and processing conditions.[12] MgO is added to the glass system to enhance charge carrier release and reduce hygroscopicity in borate glass.[13]

A borosilicate glass composite doped with Yb_2O_3 was fabricated using the melt-quenching technique and evaluated for thermoluminescence performance. The best response was found at 0.06 mol% Yb_2O_3 , and the glass showed a linear gamma dose response from 0.5 to 2 kGy, with moderate fading over time, suggesting promise for radiation detection applications.[18]

A 2024 study demonstrated that incorporating Nb_2O_5 into Eu^{3+}-doped ZnO–B_2O_3 glass modified the glass network through the formation of NbO_4 tetrahedra and NbO_6 octahedra, resulting in enhanced red emission from Eu^{3+} ions. The strongest emission comes from the $5D_0 \rightarrow 7F2/5D_0 \rightarrow 7F2$ transition under 392 nm excitation, and the emission intensity is higher for glasses with 1–5 mol% Nb_2O_5 than for the Nb_2O_5-free glass.[14] previous studies by our research group have also demonstrated the potential of glass based materials for radiation shielding application in medical setting.[15]

To the best of our knowledge, limited studies have examined the thermoluminescent characteristics of B_2O_3-CaO-Li_2O glass doped with varying MgO quantities for radiation dose measurement using a Co-60 source. The samples were prepared using the melt quenching method, a simple and cost-effective method that produces homogeneous glass. In this study, we examine the optical characteristics and TL response of this glass system to assess its suitability as a TLD.

2. MATERIALS AND METHODS

2.1 Study Material

The glass samples were prepared from high-purity reagents with the composition (80–x) B_2O_3: 10% Li_2O : 10% CaO : xMgO (x = 5, 10, 15, 20, and 25 mol%) (Alfa ALesar 99.8%). The glass samples were labelled 25 Mg, 20 Mg, 15 Mg, 10 Mg, and 5 Mg according to their MgO content. The homogenized melted mixture was molten in a pure platinum crucible in an electric-heated furnace under normal air circumstances for 1 hour at a temperature of around 1200 °C. The melted material was quenched on a preheated stainless steel mold to produce glass before being immediately transported to an annealing furnace at 350 °C for two hours. The furnace was then switched off to enable cooling to room temperature at a rate of 10 °C/min.

The same procedure was followed for each magnesium concentration, producing five cylindrical samples (3 mm in diameter and 1 mm in height). The density of glass samples was determined using Archimedes' principal, with toluene as the immersion liquid. The samples were prepared as circular discs with a radiatus of 1 cm and thickness 3 mm. The density was calculated using the following equation.

$$D\,(\mathrm{gm/cm^3}) = \left(\frac{m_a}{m_a - m_t}\right)\rho_t$$

where D is the density of the sample, m_a the weight of the sample in air, m_t the weight of the sample in toluene, and ρ_t (0.653 g/cm^3 at 20 ^{0}C) is the density of toluene. Table 1 listed the chemical compositions and codes for glass samples.

To analyze the optical properties, each glass composition was examined by FTIR spectroscopy over the 400-4000 cm^{-1} wavenumber range. Transparent pellets were prepared by thoroughly blending a small amount of powdered glass (0.004 g) with potassium bromide (0.200 g), then compressing the mixture under high pressure (15 tons) for three minutes in a vacuum. The resulting discs were measured with a JASCO 460 FTIR spectrometer. The position of the absorption peaks was measured with an accuracy of ±2 cm^{-1}.

XRD measurements were conducted on a Bruker diffractometer equipped with a Cu Kα X-ray source, and diffraction profiles were recorded over a 2θ range of 5°–90°.

The optical absorption spectrum of polished glass samples (2 mm thick) was recorded at room temperature using a Jasco V-530 UV-Vis spectrophotometer covering the wavelength range of 190-1000 nm with a resolution of 2 nm.

Energy dependence or sensitivity for different glass compositions (5Mg-25Mg) versus energy are calculated using $S(E) = (\mu_{en}/\rho)_{sample}/(\mu_{en}/\rho)_{air}$ where S(E) is the energy-dependent relative response of a bare TLD assuming negligible self-absorption and the existence of electronic equilibrium, $\mu_{en/\rho}$. The calculations are done under the assumption of electron equilibrium, monoenergetic radiation and narrow beam geometry, neglecting the self-shielding of the material as well as the fluorescence and electron escape.

Table 1. Compositions and weight fractions of the modified glass systems.

Sample code	B_2O_3	CaO	Li_2O	MgO	Density (g/cm^3)	Weight fraction
5Mg	75	10	10	5	2.554 ± 0.002	B_2O_3(0.8311), CaO(0.0893), Li_2O (0.0476), MgO(0.0321)
10Mg	70	10	10	10	2.591 ± 0.002	B_2O_3(0.7942), CaO (0.0914), Li_2O (0.0487), MgO(0.0657)
15Mg	65	10	10	15	2.633 ± 0.004	B_2O_3(0.7556), CaO(0.0936), Li_2O (0.0499), MgO(0.1009)
20Mg	60	10	10	20	2.677 ± 0.003	B_2O_3(0.7149), CaO(0.0960), Li_2O (0.0511), MgO(0.1379)
25Mg	55	10	10	25	2.721 ± 0.002	B_2O_3(0.6722), CaO(0.0985), Li_2O (0.0525), MgO(0.1769)

The prepared glass samples were irradiated with a cobalt gamma source at a dose of 12.38 Gy and a dose rate of 13.29 Gy/h. Thermoluminescence (TL) intensity was measured using a Harshaw 4500 TLD reader (Harshaw, Cleveland, USA). Samples were exposed to heating from 50 ^{o}C to 400 ^{o}C at a rate of 5 ^{o}C/s under nitrogen flow. The heating applied during the readout process (TLD measurements) was found to be sufficient to eliminate any residual dose from the samples.

3. RESULTS AND DISCUSSION

3.1 X-ray diffraction (XRD)

XRD patterns of all glass samples of B_2O_3-CaO-Li_2O modified with MgO are presented in Fig.1. The XRD patterns show that display only two distinct halo peaks which related to borate glass system.

Fig.1 XRD pattern of B_2O_3-CaO-Li_2O glass samples doped with different MgO concentrations.

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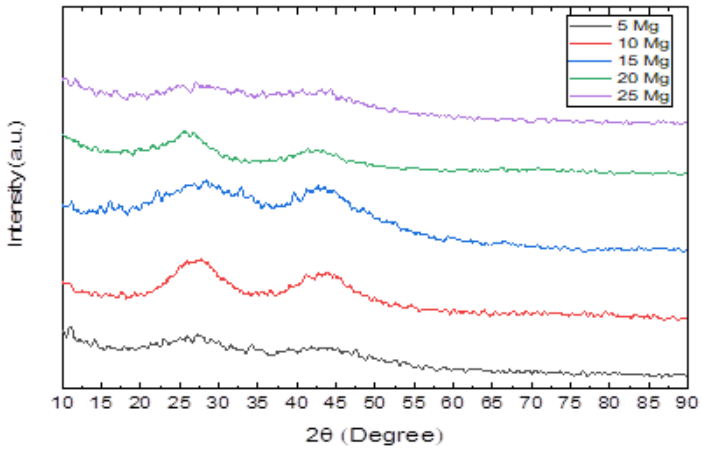


Fig.1 XRD pattern of B_2O_3-CaO-Li_2O glass samples doped with different MgO concentrations.

3.2 FTIR spectroscopy

FTIR spectra provide information about the structural units in the glass dosimeter matrix, as shown in Fig.2. The vibration modes of the borate group may be classified into different regions based on their characteristics. Asymmetric stretching vibrations of BO_3 units are observed in the region of 1200-1600 cm^{-1}, while stretching vibrations of BO_4 units occur between 800-1200 cm^{-1}. Additionally, bending vibrations appear in the range 600 and 800 cm^{-1}.

The low wavenumber region is principally connected with the vibrations of metal cations (Li^+, Ca^{2+}, and Mg^{2+}) inside the glass network, as well as the bending vibrations of B-O-B linkages. MgO acts as a network modifier. When MgO is incorporated into the B_2O_3 network, it breaks B-O-B bridging bonds, converting BO_3 triangles into BO_4 tetrahedra or creating non-bridging oxygens (NBOs). Since electrons are more loosely bound to NBOs than to bridging oxygens, less energy is required for excitation, leading to reduction of the band gap.

The assignment of the vibrational bands for the prepared glass dosimeter is shown in Table 2. In all glass samples, increasing the MgO content does not greatly impact the structure of glass dosimeter samples, as shown by the absence of new bands.

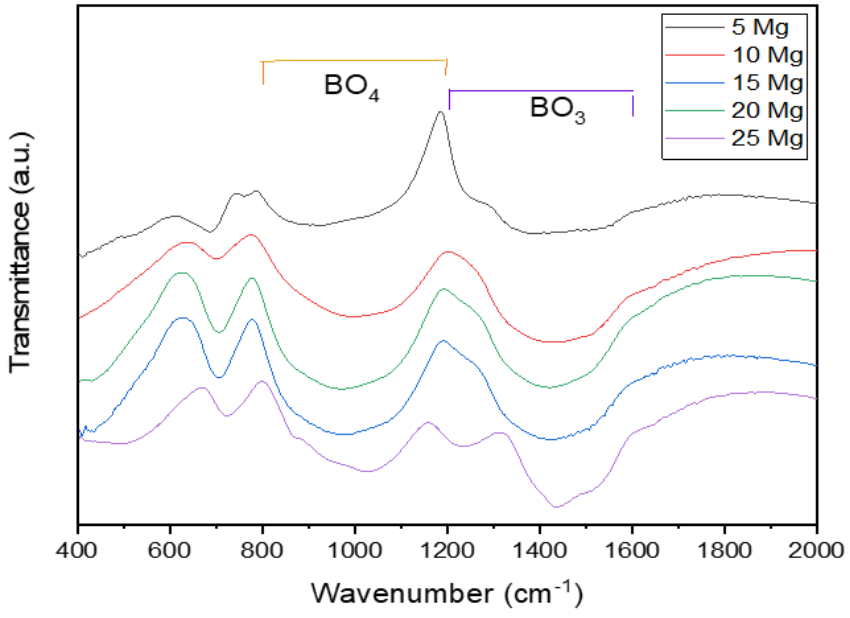


Fig.2 FTIR spectrum of Li2O-CaO-B2O3 glass doped with MgO.

Table 2: Assignment of infrared bands in the spectra of different glass samples

Wavenumber (cm^{-1})	Band Assignment	References
700-716	B-O-B linkage in the borate network	16
1000	Stretching vibration of the O-B bonds of BO_4 units in tri-, tetra- and pentaborate groups	17
1432	Asymmetric streteching vibration of the B-O band of BO_3 group	16

3.3 Measurements of UV-VIS spectroscopy

Fig.3 illustrates the UV-Visible spectrum of $10Li_2O+10CaO+(80–x)\ B_2O_3$ glass samples modified with xMgO where x=5, 10, 15, 20, 25 mole%. With increasing MgO concentration, the absorbance of samples increases slightly, and the broad band shifts toward higher wavelengths, indicating a decrease in the optical band gap.

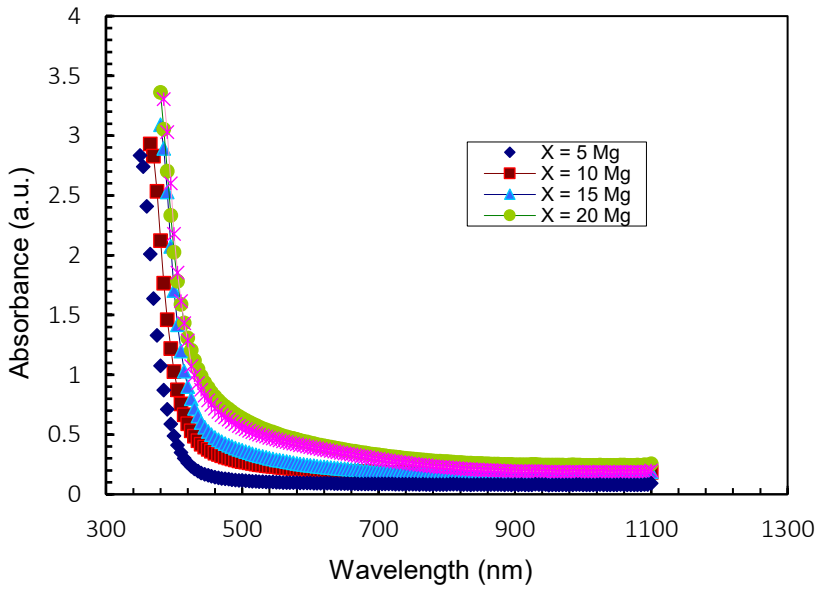


Fig. 3 Absorption spectrum of Li_2O-CaO-B_2O_3- MgO glass-modified system.

Atomic bonding has an effect on the optical bandgap energy (E) for indirect transitions. Tauc plots (Fig. 4) were used to determined optical band gap values. The linear region of $(\alpha h\nu)^{1/2}$ versus $h\nu$ was extrapolated to the energy axis. The addition of MgO led to decreases in band gap energy from 2.91 to 2.65 eV due to structural changes in the glass. Increased MgO concentrations (from 5 to 25 mol%) can increase localization by producing charge distribution defects and bringing oxygen ions positioned nearer to the upper region of the valence band, resulting in a higher number of donor centers in the glass samples matrix. Increased donor centers cause a reduction in energy bandgap. As the optical band gap increases, the TL intensity also increases; therefore, most materials used in dosimetric applications have a high energy band gap[19].

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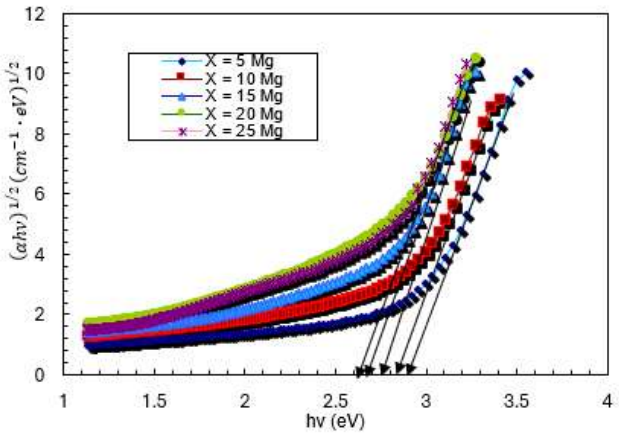


Fig.4 Tauc plot for indirect optical band gap of Li_2O-CaO-B_2O_3- MgO glass-modified.

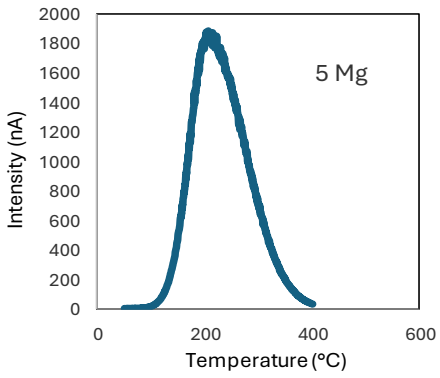
3.4 TL Glow Curves

The glow curve is obtained by measuring luminescence as a function of temperature. Fig. 5 illustrates the characteristic TLD glow curves of lithium calcium magnesium borate samples after 12.38 Gy gamma exposure, measured at a continuous heating rate of 5 °C/s.

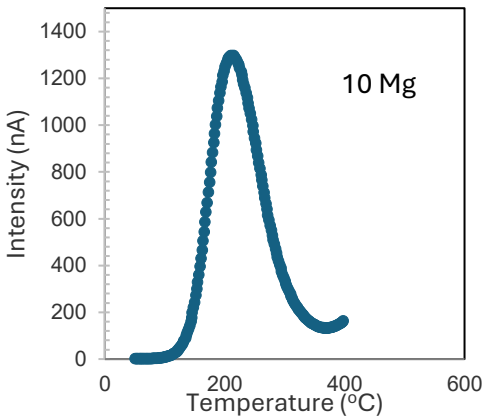
The glow curve of the glass system exhibits one broad peak, which may be composed of multiple overlapping glow peaks. Each peak is representative of a particular trap inside the lattice. When the sample is heated during readout, electrons trapped in shallower traps are released faster than those in deeper traps.

The peak occurring at a relatively high temperature around 205 °C suggests that the traps are sufficiently deep to avoid significant fading at room temperature. As the MgO concentration increases to 15 Mg, 20 Mg, and 25 Mg samples, the peak intensity shows a significant reduction.

For 12.38 Gy gamma exposure, the highest TL intensity was observed in lithium calcium borate modified with 5 mol% Mg, and with increasing Mg concentration, the TL intensity decreases. The reduction in TL intensity seen with increasing MgO concentration might be related to more non-radiative recombination centers or changes in trap distribution.



(a)



(b)

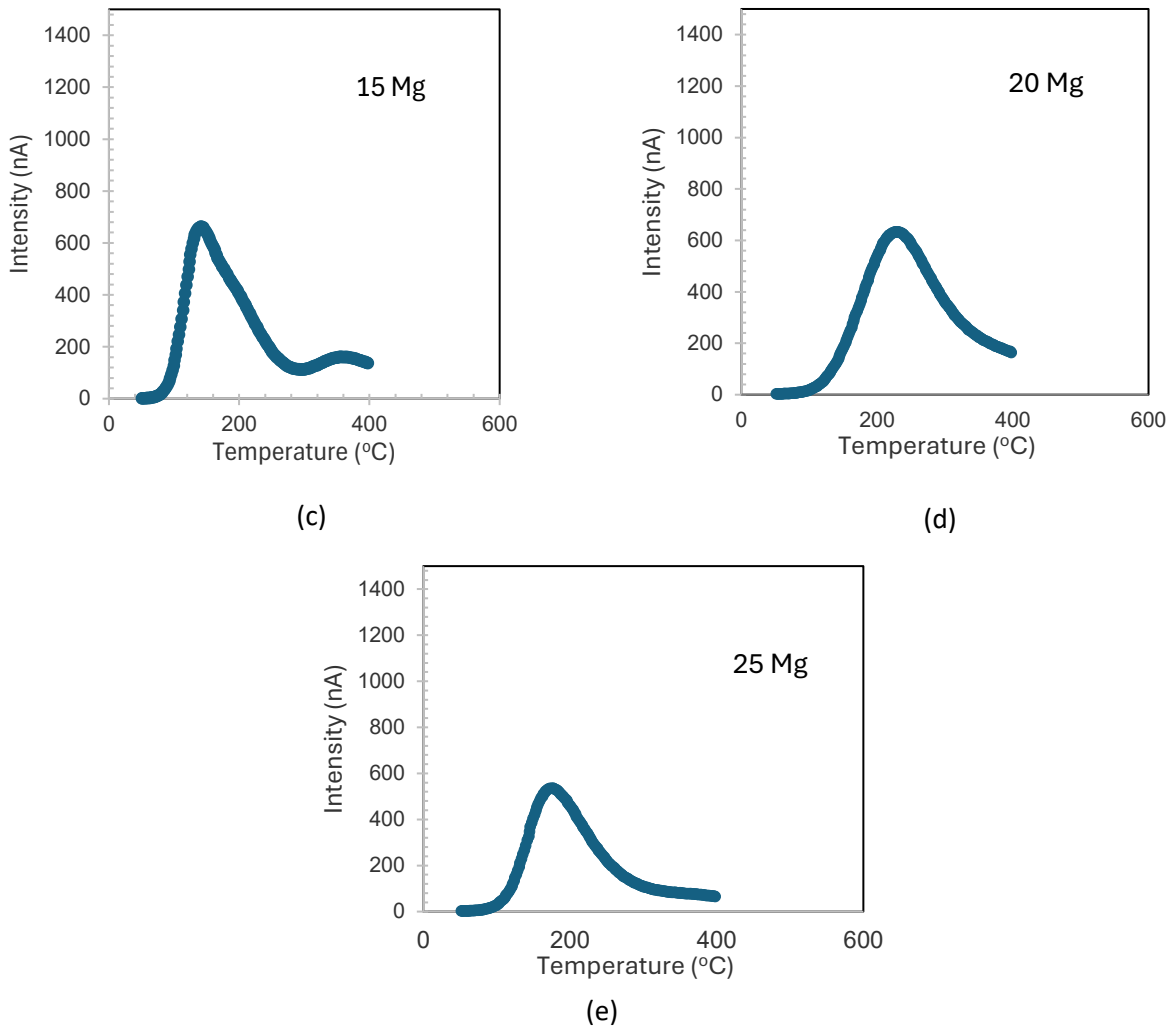


Fig. 5. glow curves of Li2O-CaO-B2O3 samples modified with MgO at varying concentrations (irradiated dose: 12.38 Gy): (a) 5Mg, (b) 10Mg, (c) 15Mg, (d) 20Mg, and (e) 25Mg.

3.5 Dose response

To investigate the dose dependence of the TL response, glass-disk samples were irradiated with gamma doses ranging from 0.4 to 12.38 Gy. Three chips with identical composition and structure were irradiated at each dose , and the average TL signal was calculated and used to evaluate the dose-response behavior. Fig. 6(a) and 6(b) present the glow curves of the 5Mg system irradiated at different gamma doses and dose linearity curve, respectively.

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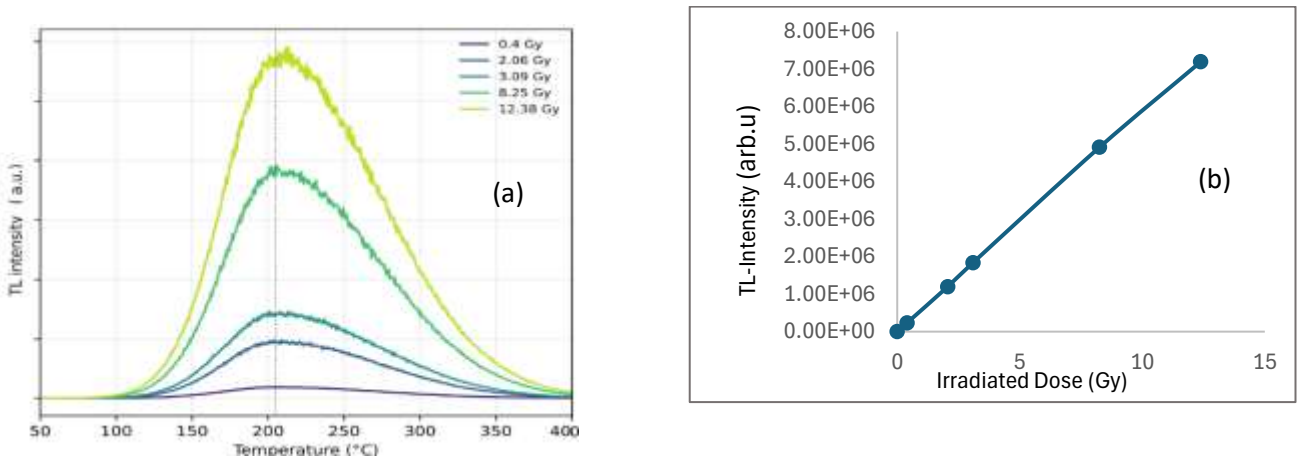


Fig. 6 (a) The dose-response for the glass system 5Mg. (b) Dose-linearity fitting curve (area under curves versus γ-dose) extracted from Fig. 6 (a).

The energy dependence of the glass samples was evaluated by calculating the relative response, S(E), as a function of photon energy, as shown in Fig.7. For all glass compositions, S(E) increased with increasing MgO content and reached a broad maximum at approximately 52 keV before decreasing at higher photon energies. The 25Mg detector exhibited the highest response, which may be attributed to its higher effective atomic number. In contrast, the 5Mg sample showed the lowest energy dependence, a desirable characteristic for medical dosimetry applications. Determination of S(E) is useful for selecting suitable medical applications and for designing appropriate energy filters or correction methods for the TL material.

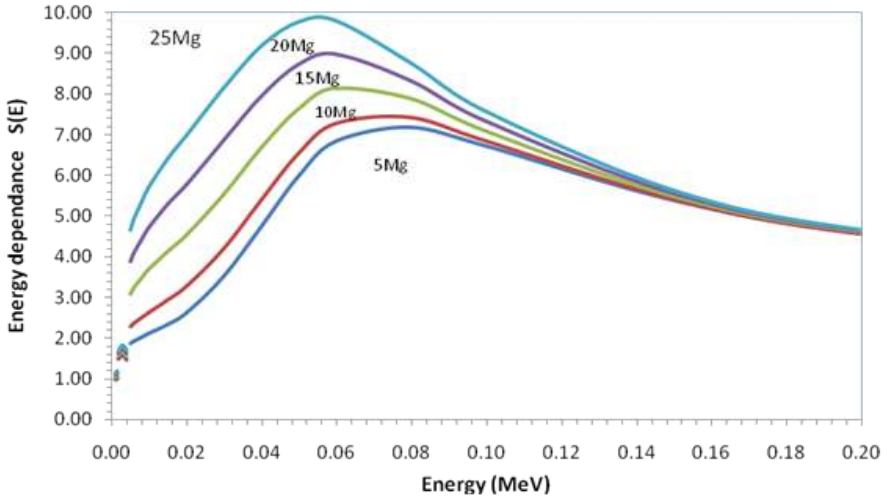


Fig. 7. S(E) as a function of photon energy for 5-25Mg glass detector.

The fading behavior of the TL signal is important when TL phosphors are used for ionizing-radiation monitoring. The 5Mg glass sample exhibited the greatest signal decrease during the initial 48 h at room temperature, after which the TL signal gradually stabilized and remained relatively stable for up to 3240 h (see Fig. 8).

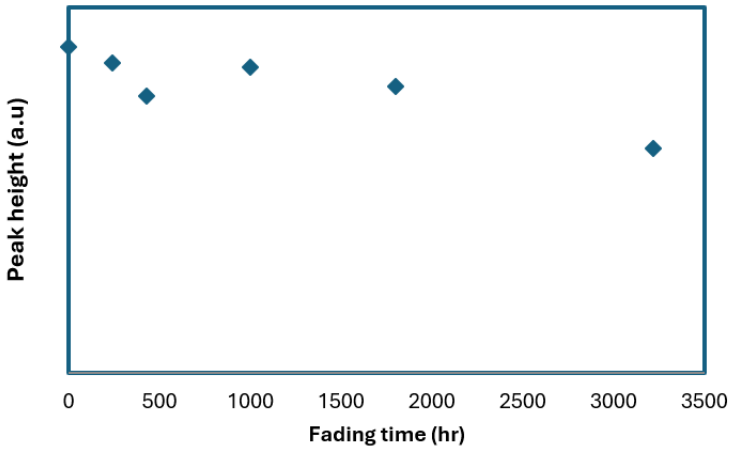


Fig. 8 The fading effect of glow curve peak pre-exposed at 12.38 Gy and read-out after 0, 242, 427, 1000, 1800 and 3240 hrs.

4. CONCLUSION

$Li_2O–CaO–B_2O_3$ transparent glass samples modified with different MgO concentrations were successfully synthesized using the melt-quenching technique. The synthesized glasses were characterized using XRD, FTIR, and UV–visible spectroscopy. The XRD patterns confirmed the amorphous nature of all samples, while the FTIR spectra revealed the presence of BO_3 and BO_4 structural units. The optical band gap decreased from 2.91 to 2.65 eV with increasing MgO content, which may be attributed to the formation of non-bridging oxygens. TL measurements after 12.38 Gy gamma irradiation demonstrated that the glass system produces a broad TL glow peak, suggesting overlapping trap-dependent processes. The TL glow-peak intensity also decreased with increasing MgO concentration, whereas the sample containing 5mol% MgO showed the highest TL intensity. The trap responsible for TL emission appears to be sufficiently deep around 205 °C, which helps reduce room-temperature fading. The 5Mg phosphor exhibited an excellent linear TL response over a broad gamma-dose range of 0.4–12.38 Gy. The fading study showed an increase in signal decay after 48 hours, followed by stabilization for up to 3240 hours (135 days). The 5Mg glass detector exhibited the lowest energy dependence among the investigated samples, making it highly suitable for medical dosimetry applications.

AUTHOR CONTRIBUTIONS

Hani Alhazmi: Conceptualization, Methodology, Investigation, Data interpretation, Writing Original Draft: Samia Faisal O. Aboushoushah: Supervision, Writing—Review & Editing: Hossam Donya: Methodology, Contributed to the study design and interpretation of data.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

FUNDING

NO FUNDING.

ETHICS STATEMENT

ETHICAL APPROVAL WAS NOT REQUIRED FOR THIS STUDY.

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